

DERWENT-ACC-NO: 1999-572408

DERWENT-WEEK: 200337

COPYRIGHT 1999 DERWENT INFORMATION LTD

TITLE: Cryogenic treatment of a sintered tungsten carbide
button insert for a drill

INVENTOR: ELSBY, J

PATENT-ASSIGNEE: ELIKON PTY LTD[ELIKN]

PRIORITY-DATA: 1998AU-0002303 (March 12, 1998) , 1998AU-0001992
(February 25,
1998)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES
MAIN-IPC			
AU 760484 B	May 15, 2003	N/A	000 C21D 001/613
AU 9918399 A	September 9, 1999	N/A	015 C21D
001/613			

APPLICATION-DATA:

PUB-NO	APPL-DESCRIPTOR	APPL-NO	APPL-DATE
AU 760484B	N/A	1999AU-0018399	February 23, 1999
AU 760484B	Previous Publ.	AU 9918399	N/A
AU 9918399A	N/A	1999AU-0018399	February 23, 1999

INT-CL (IPC): C21D001/613

ABSTRACTED-PUB-NO: AU 9918399A

BASIC-ABSTRACT:

NOVELTY - Tungsten carbide part sintered with 6 wt.% cobalt binder is treated with boron, and treated cryogenically with a dry nitrogen or inert gas atmosphere. It is cooled at a rate where internal stresses are dissipated, to

-100 deg. F and then gradually cooled further to a transition temperature (approximately -300 deg. F), and held for 10-15 hours before warming. It is maintained clear of liquefied refrigerant gas. Cooling and warming is at 1 deg. F/minute. Warming is interrupted at -110 deg. F for ultrasonic stress relief.

USE - Treating a sintered tungsten carbide part used for the production of tools, inserts, dies, etc., especially a button insert (all claimed) for drill used in rock drilling.

ADVANTAGE - Wear resistance of the part is improved, and the cooling/warming rates prevent any thermal shock damage occurring.

CHOSEN-DRAWING: Dwg.0/3

TITLE-TERMS: CRYOGENIC TREAT SINTER TUNGSTEN CARBIDE BUTTON INSERT DRILL

DERWENT-CLASS: L02

CPI-CODES: L02-A; L02-F03; L02-H02A;

SECONDARY-ACC-NO:

CPI Secondary Accession Numbers: C1999-167211

I
METHOD FOR TREATMENT OF SINTERED CARBIDES

This invention relates to a method for treatment of sintered carbides.

This invention has particular application to a method for treatment of sintered tungsten carbide buttons for the drilling industry to increase wear resistance, and
5 hereinafter this invention will be described in terms of this application. However, it is envisaged that methods for treatment of carbides in accordance with the present invention may find other applications such as treatment of other carbide structures such as dies, metal working tools or the like.

In rock drilling a common form of drill bit is the hammer drill bit in which a steel
10 body has tungsten carbide inserts in the cutting face. This bit is then attached to a percussion motor which in conjunction with rotation of the bit serves to break the rock. In a lot of hard abrasive drilling conditions there can be extreme carbide wear due to the carbides being ground away on the abrasive cutting face of the hole. Due to this extreme wear the drilling penetration rate will reduce as the wear increases. If
15 the bit is not removed from the hole and drilling continues the operator runs the risk of bit failure through a number of mechanisms. A process called hot isostatic pressing is now the current accepted industry standard for inserts used in the mining industry. This process compresses the structure and reduces voids in the grain structure to provide a more homogenous grain structure which improves the
20 mechanical properties.

As the carbides wear the rounded appearance of the cutting surface will flatten out thus vastly increasing the load on the carbide due to its reduced ability to break the rock while the output of the drill has not changed. This increase in carbide load can ultimately result in carbide failure. Further, due to the increased load on the
25 carbide there is a corresponding increase in the load on the body of the bit which

houses the carbides. Even if the carbides do not fail there can be a failure of the bit body through cracks forming and ultimately portions of the head breaking off.

Apart from the obvious cost of replacing the failed bit there can also be the problem that another hole must be drilled due to the obstruction caused in the hole by a broken portion of the bit. Due to the problems mentioned with worn carbides it is necessary for the operator to remove the bit from the hole to sharpen the carbides. In some areas it is common that the intervals between sharpening is as low as 10 metres. As a result it has been important to provide a tungsten carbide insert which exhibits a high degree of wear resistance.

There are many examples of procedures that are used to increase wear resistance of carbides. One such example is used in the metal cutting tool industry is titanium nitride (TiN) coatings. The disadvantage with coatings when used in rock drilling inserts is that, hard as they are, as the coating wears away the benefits gained by coating are lost. Another example is Boron infusion to the surface of the carbide that increases the toughness (resistance to brittle failure) and allows inserts sintered from finer grades of carbide to be used. Fine grained carbides exhibit better wear characteristics but are susceptible to brittle failures in the absence of such boron treatment. However, it is speculated that the spread of grain sizes in the sintered product limits the effectiveness of boron treatment. Commercially available tungsten carbide inserts are available with different grain structures the size and configuration of these structures has a marked effect on the mechanical properties in particular wear and impact strength.

Cryogenics have been used in the past as a treatment which increases wear resistance for steels, particularly heat treated high Carbon steels. However, conventional wisdom would have it that relatively brittle homogenous materials such

as sintered tungsten carbides would present problems due to the thermal shock which occurs when a component is suddenly subjected to a sharp reduction in temperature. It would be expected that this would sometimes lead to failure of the component through cracking. In any case the cryogenic treatment of sintered carbides has not been reported as beneficial in terms of promoting wear resistance.

It is thus an object of the present invention to provide a method for treatment of sintered carbide parts which will be reliable and efficient in use and to produce parts which overcome at least one of the disadvantages of the prior art described above.

10 With the foregoing and other objects in view this invention in one aspect resides in a method for treatment of sintered carbide parts including the step of cryogenic treatment of the parts.

Preferably, the method of the present invention is used to increase the wear resistance of standard commercially available tungsten carbide inserts. It is 15 speculated that the cryogenic treatment of sintered carbide parts results in further improved homogeneity by contraction of the grain structure through temperature reduction and expansion back to a more uniform structure by heating back to ambient temperature. This process also appears to serve to relieve inherent stresses in the structure.

20 The sintered carbide parts may be of any type used for the production of tools, inserts, dies or the like. For example, such components may be formed of tungsten carbide sintered with a cobalt binder material and formed by hot isostatic pressing. If desired, the parts may be boron treated.

The cryogenic treatment is preferably performed in a relatively dry, inert 25 atmosphere. For example, the parts may be cooled in a dry nitrogen or inert gas

atmosphere. The parts are preferably kept clear of any liquid refrigerant that may encourage thermal shock. The parts may be progressively cooled from room temperature. Alternatively, the parts may be relatively rapidly cooled to a temperature and at a rate within the ability of the part to dissipate internal stresses.

- 5 For sintered tungsten carbide button inserts it has been established that the parts will cope with cooling in a refrigerated atmosphere maintained at about -100°F .

The cryogenic process may then amount to gradual reduction of the chamber temperature to a transition temperature that may be determined experimentally for different sintered carbide compositions. In the case of sintered tungsten carbide
10 parts comprising tungsten carbide particles in a binder comprising 6% by weight cobalt, and being conventionally treated with boron, the experimentally determined temperature was in the region of about -300°F .

The cooling of the chamber may be achieved by any suitable means. For example, the temperature of the initial rapid cooling if used may be achieved by heat
15 exchange means such as a Carnot cycle refrigeration plant. Further progressive cooling may then be advantageously done using liquid gases to cool the chamber atmosphere. Alternatively, liquid gases may be used for the whole cycle. Any suitable liquid gas may be used consistent with the gas having a boiling point low enough to cryogenically treat the particular carbide material. For example, non-toxic,
20 non-flammable liquid gases such as nitrogen and helium may be used, with liquid nitrogen preferred on the ground of cost.

The temperature of the cooling chamber is preferably controlled such that the progressive drop in temperature of the parts is sufficiently slow to allow stresses to dissipate. It has been experimentally determined that rates in the region of $-1^{\circ}\text{F}/\text{min}$
25 are satisfactory for tungsten carbide drill bit buttons. Accordingly, the temperature

may be controlled by any suitable means such as controlling the supply of refrigerant gas or the like. Preferably, the control means comprises automatic control means such as by a computer interface which controls the introduction of liquid nitrogen to the refrigerated chamber to achieve, for example a temperature drop of 1°F per
5 minute until a temperature of about -300°F is reached, in the case of tungsten carbide buttons.

The cryogenic process preferably includes a hold or dwell time that may be experimentally determined in order to equilibrate the structure. For example, the temperature may be held at the preferred about -300°F for a period of time
10 comprising the temperature equilibration time depending on the volume of carbides to be treated plus an experimentally determined hold time. In the case of the preferred tungsten carbide buttons, the minimum hold time is preferably in the region of 10-15 hours.

The equilibrated parts may then be warmed to room temperature. Preferably
15 the parts are warmed at a sufficiently slow rate to allow thermal stresses to dissipate. For example, in the case of tungsten carbides it has been found advantageous to warm the parts at a similar rate to the preferred rate of cryogenic cooling, or by about
+1°F per minute.

The parts may be progressively warmed to room temperature. However, it
20 has been found advantageous to interrupt the warming at a temperature whereby stress relief may be done such as ultrasonic stress relief. For example in the case of tungsten carbide parts, the controlled temperature rise may be arrested at about -110°F whereupon further treatment can be then carried out such as by ultrasonic stress relieving.

25 Whilst not wishing to be bound by theory, it is speculated that the cryogenic

treatment of sintered carbide components further improves homogeneity by contraction of the grain structure through temperature reduction and expansion back to a more uniform structure by heating back to ambient temperature. This process also serves to relieve inherent stresses in the structure.

5 The invention will hereinafter be further described with reference to a preferred embodiment thereof as illustrated in the following example and the accompanying Figures wherein:

FIG. 1 is an electron micrograph of a sintered tungsten carbide surface in an as-produced condition;

10 FIG. 2 is an electron micrograph of a carbide surface as per FIG. 1, with cryogenic treatment down to -100°F in accordance with the present invention, and

FIG. 3 is an electron micrograph of a carbide surface as per FIG. 1, with cryogenic treatment down to -300°F in accordance with the present invention.

15 EXAMPLE 1

Carbide hammer drill bit buttons were prepared in the conventional manner by hot isostatic pressing of a composition comprising tungsten carbide particles and 6% by weight of cobalt. The sintered buttons were then post treated with boron in the conventional manner.

20 A refrigerated chamber is provided comprising an insulated housing having a cooling element therein. The cooling element is associated with a supply of liquid nitrogen regulated by a supply valve operated by an electronic controller responsible to a thermostat. The controller is programmable such that a cooling regime may be imposed on the chamber.

25 The chamber was cooled to and maintained at -112°F to equilibrate the

chamber. The buttons were placed in the chamber, equilibrated at -112°F and then gradually cooled to -300°F . This is achieved by a computer interface which controls the introduction of liquid nitrogen to the refrigerated chamber to achieve the desired cooling rate of about 1°F per minute until the required temperature of -300°F is reached. The cooling regime was plotted as a function of time in accordance with the following table:

TABLE 1

	Hour	Temperature $^{\circ}\text{F}$
	0	-50
10	3.5	-100
	5.25	-214
	6	-214
	6.5	-240
	7.75	-270
15	8.5	-300

The components were then equilibrated and held at -300°F for a total of 16.5 hours, followed by a 10 hour program of temperature rise to -100°F , whereupon the parts were ultrasonically stress relieved. The parts were then progressively warmed to room temperature.

Buttons prepared in accordance with the foregoing embodiment exhibited superior wear resistance to control buttons not cryogenically treated. By way of illustration of the apparent effect on the structure of the carbide surface, FIGS. 1 to 3 illustrate carbide surfaces at 1500x magnification and comprising an untreated boron infiltrated carbide surface, a carbide surface cryogenically treated to -100°F , and a carbide surface treated in accordance with the present example, respectively.

In the figures it can be seen that the structure of the carbide surface of FIG. 3 is more even in particle size and distribution than either of the surfaces of FIGS. 1 and 2. The surface of FIG. 2 is marginally but not remarkably improved over that of the surface of FIG. 1.

5 An example of each type of sintered carbide downhole hammer buttons, that is, conventionally boron treated, boron treatment with -100°F cryogenic treatment and boron treatment with -300°F cryogenic treatment were each subjected to abrasive wear by way of bearing of the hemispherical button surface against a diamond grinding wheel for 3 minutes. The buttons are notionally 63.5 g wt. The
10 table of actual weights, weight after grinding, weight lost as a percentage, and relation to the untreated button as a standard is presented as Table 2 hereinafter.

Table 2

Treatment	Initial Weight	Final Weight	%wt Loss	Relative %wt Loss
none	63.885g	62.789g	1.72	N/A
-100°F	63.542g	62.481g	1.67	-2.9
-300°F	63.462	62.477g	1.55	-9.88

The improvement in wear resistance exhibited by buttons in accordance with
15 the abovedescribed embodiment comprising cryogenic treatment at -300°F represents an improvement in service in a downhole hammer of from 50 to 100% of service life, depending on ground conditions.

It will of course be realised that while the foregoing has been given by way of illustrative example of this invention, all such and other modifications and variations
20 thereto as would be apparent to persons skilled in the art are deemed to fall within the broad scope and ambit of this invention as defined in the claims appended hereto

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. A method for treatment of sintered carbide parts including the step of cryogenic treatment of the parts.
2. A method according to claim 1, wherein the part is a sintered tungsten carbide part.
3. A method according to claim 2, wherein said sintered tungsten carbide part is selected from any type used for the production of tools, inserts, dies or the like.
4. A method according to any one of the preceding claims, wherein said part is of tungsten carbide sintered with a cobalt binder material and formed by hot isostatic pressing.
5. A method according to claim 4, wherein the part is boron treated.
6. A method according to any one of the preceding claims, wherein said cryogenic treatment is performed in a substantially dry, inert atmosphere.
7. A method according to claim 7, wherein said atmosphere is selected from a dry nitrogen or inert gas atmosphere.
8. A method according to any one of the preceding claims, wherein the part is maintained clear of any liquefied refrigerant gas.
9. A method according to any one of the preceding claims, wherein the part is may be progressively cooled from room temperature.
10. A method according to any one of claims 1 to 8, wherein the part is relatively rapidly cooled to a temperature and at a rate within the ability of the part to dissipate internal stresses.
11. A method according to claim 10, wherein said part is a sintered tungsten carbide button insert and wherein said rapid cooling is by means of a

refrigerated atmosphere maintained at about -100°F .

12. A method according to any one of the preceding claims, wherein said cryogenic process includes cooling the part in a chamber by gradual reduction of the chamber temperature to a transition temperature characteristic of said sintered carbide compositions.
13. A method according to claim 12, wherein said part is a sintered tungsten carbide part comprising tungsten carbide particles in a binder comprising 6% by weight cobalt, and being conventionally treated with boron, and wherein said characteristic transition temperature is in the region of about -300°F .
14. A method according to any one of the preceding claims, wherein said part is cryogenically treated by cooling with a gas selected from helium and the vapour of liquid nitrogen.
15. A method according to any one of the preceding claims, wherein the temperature of the cooling chamber is controlled such that the progressive drop in temperature of the parts is sufficiently slow to allow stresses to dissipate.
16. A method according to claim 15, wherein said part is a sintered tungsten carbide part and wherein said cooling rate is in the region of $-1^{\circ}\text{F}/\text{min}$.
17. A method according to any one of the preceding claims, wherein the cryogenic process includes a hold or dwell time at the transition temperature of the material of the part in order to equilibrate the structure.
18. A method according to claim 17, wherein the part is a sintered tungsten carbide part and wherein the hold time is 10 to about 15 hours.
19. A method according to any one of the preceding claims, and further including the step of warming the cryogenically treated part room temperature at a rate

selected to allow thermal stresses to dissipate.

20. A method according to claim 19, wherein said warming rate is selected to be normal to the cryogenic cooling rate.
21. A method according to claim 20, wherein the part is a sintered tungsten carbide part and wherein said warming rate is about +1°F per minute.
22. A method according to any one of claims 19 to 21, wherein said warming is interrupted at a temperature whereby stress relief may be done.
23. A method according to claim 22, wherein said stress relief is by ultrasound.
24. A method according to claim 23, wherein the part is a sintered tungsten carbide part and wherein the interruption of the warming is at about -110°F.

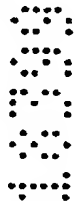
DATED THIS TWENTY-FOURTH DAY OF FEBRUARY, 1999.

ELIKON PTY LTD

(ACN 060 738 909)

by their Patent Attorneys

PIZZEYS



ABSTRACT

Carbide hammer drill bit buttons were prepared by hot isostatic pressing tungsten carbide particles and 6% by weight cobalt, the sintered parts being post treated with boron. An insulated housing includes cooling elements cooled by liquid nitrogen regulated by a supply valve operated by a programmable electronic controller responsible to a thermostat. The chamber is equilibrated at -112°F , the buttons are inserted and equilibrated at -112°F , then gradually cooled to -300°F at a cooling rate of about -1°F per minute, equilibrated and held at -300°F for a total of 16.5 hours, followed by a 10 hour program of temperature rise to -100°F , whereupon the parts were ultrasonically stress relieved. The parts were then progressively warmed to room temperature.

1/2

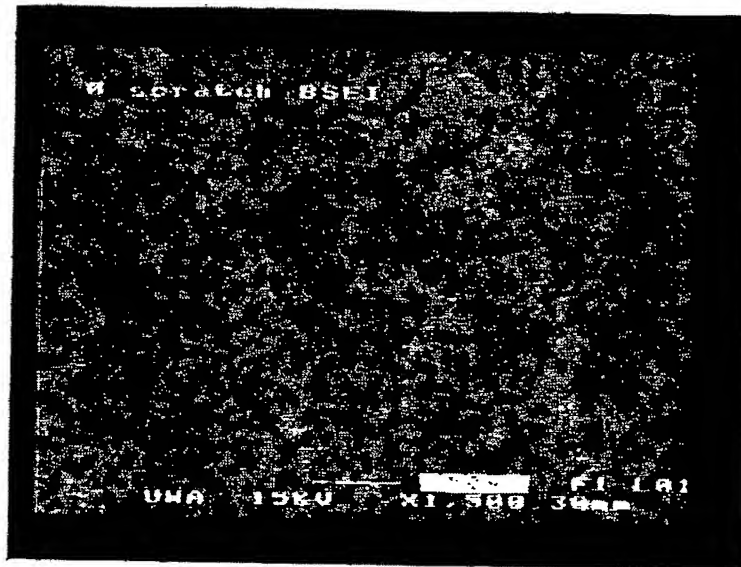


FIG 1

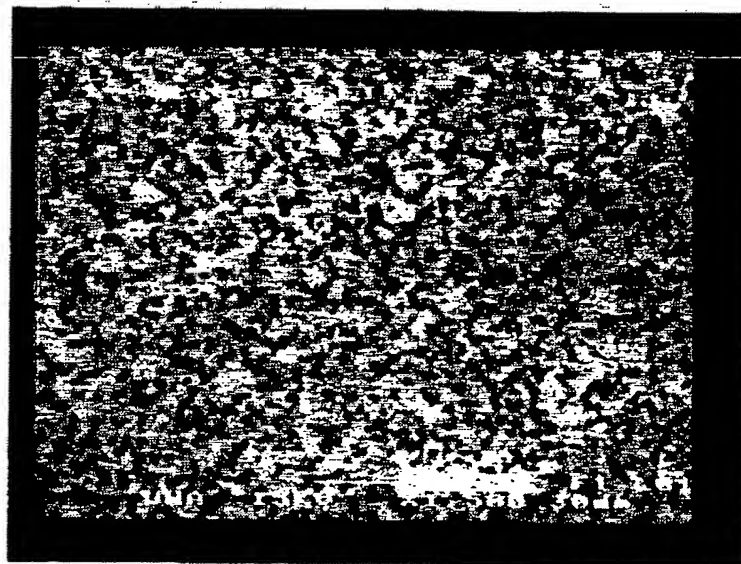


FIG 2



2/2

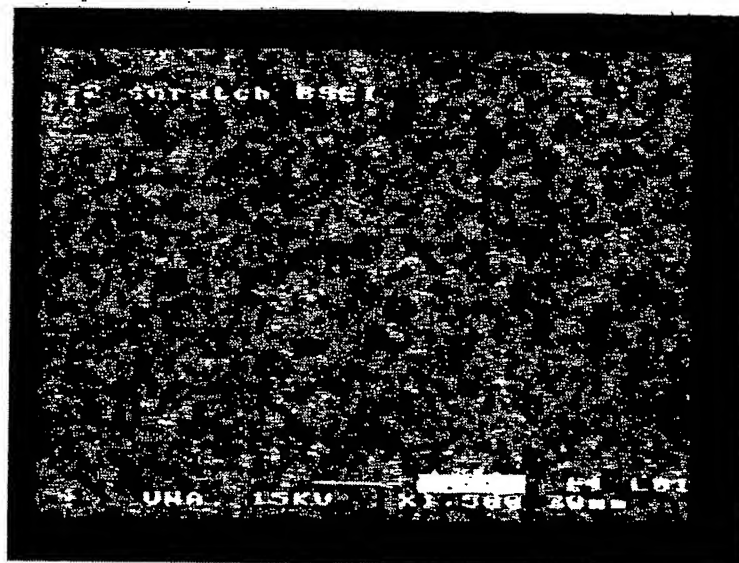


FIG 3

